

SAMPLE ENVIRONMENTS AT A SYNCHROTRON

K. Kiefer (HZB), S. Carlson (MAXIV), M. Bartkowiak (PSI)

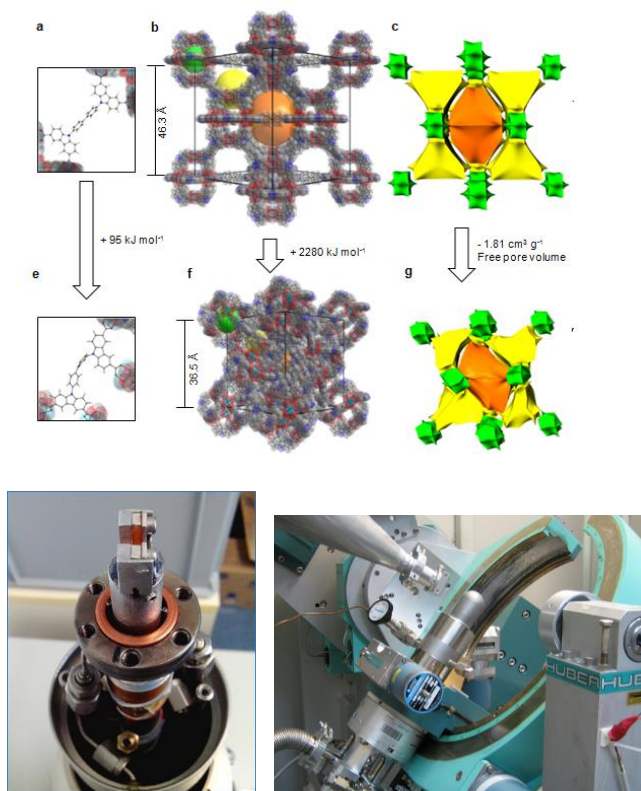
School on Synchrotron Light Sources and their
Applications | (SMR 3611), 9 December 2021



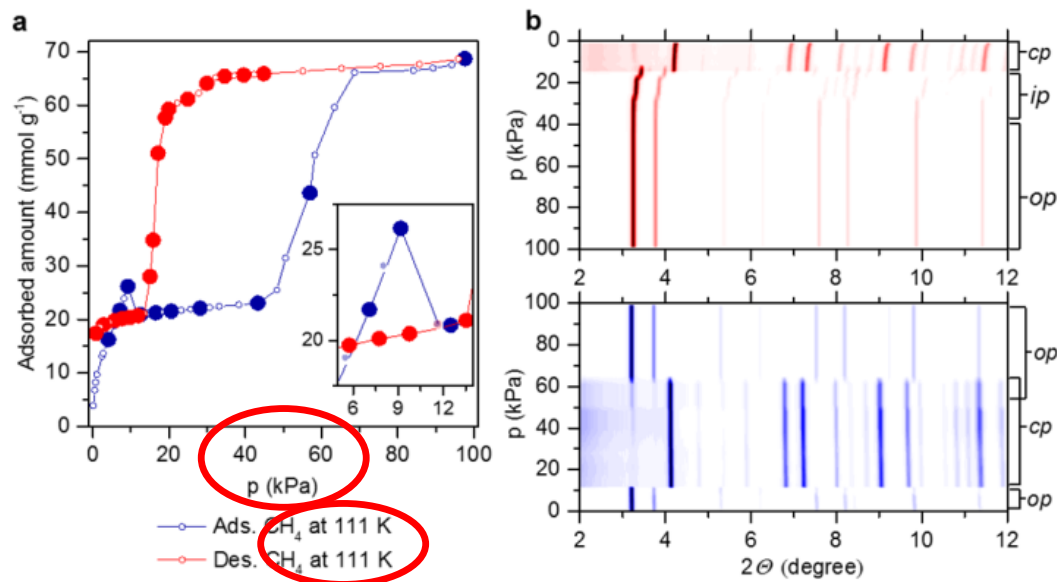
What is sample environment?

- Physical parameters: temperature, pressure, magnetic field, electric field, ...
- In-situ / in-operando experiments: catalysis, energy storage, ...
- Sample positioning: nano-positioning, rotation, ...
- Sample mounting
- Everything that effects the sample - minus the actual measurement

Motivation: Pressure Amplifying Framework with negative gas adsorption transitions



In-situ gas adsorption in combination with WAXS at beamline KMC-2



S. Krause et al. Nature 532, 348 (2016)

What is sample environment? knowledge & people

Scientists



Engineers



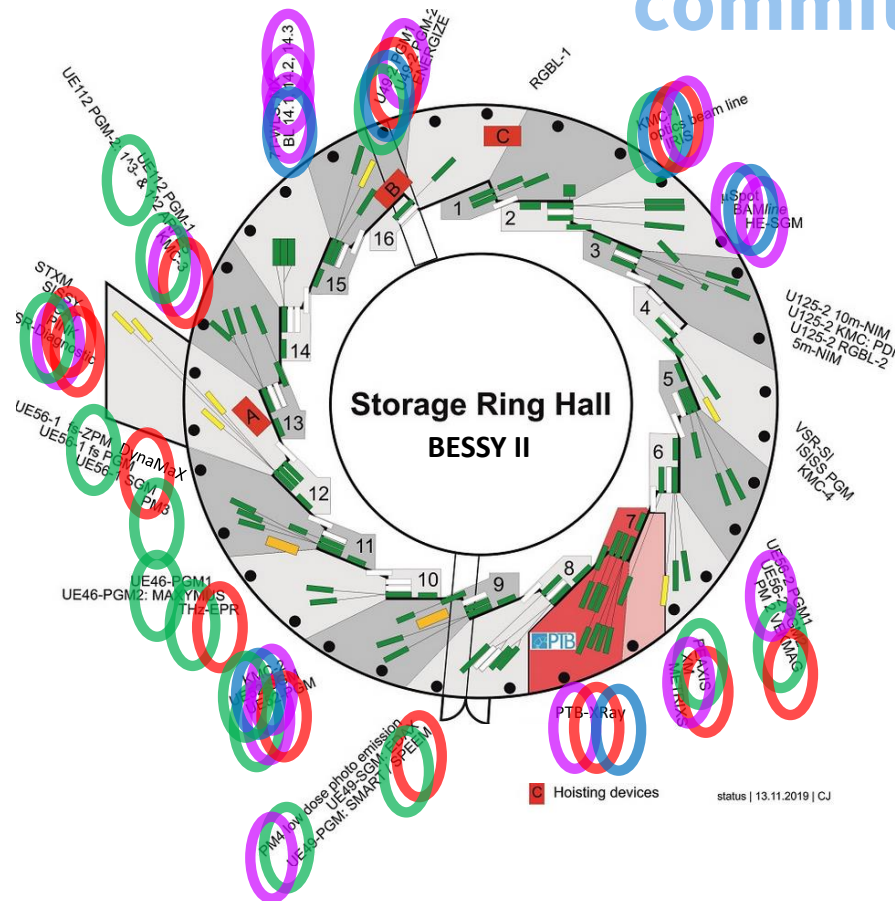
Technicians



Students



What is sample environment? commitment!



Technical developments

Optimization and repair
(of existing SE equipment)

Development (of novel SE)

Consulting (of new beamline projects)

User service

Provision and setup (of modular and complex SE)

Overview

- What is sample environment?
- General challenges
- Sample environment for T, B, p
- In-situ, in-operando & other complex things
- Sample positioning, mounting & automation

- Sample environment at MAX IV (S. Carlson, MAX IV)

- The International Society for Sample Environment (M. Bartkowiak, PSI)

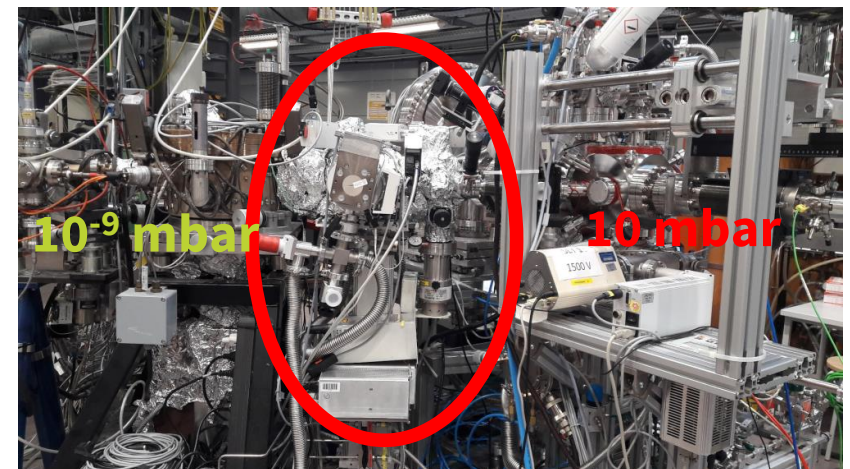
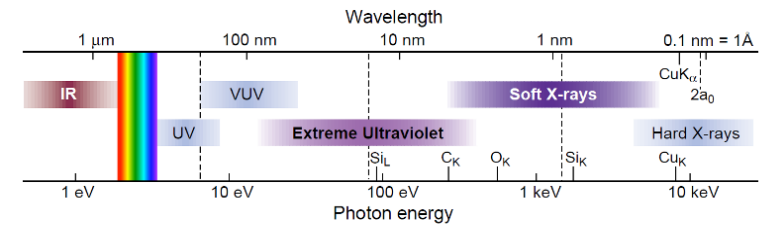
General challenges: vacuum & sample

Experiments

- Spectroscopy (detection of e^- , $h\nu$, ions): UHV necessary
- X-ray Microscopy, soft matter, biological tissue: UHV necessary
- Resonant, non-resonant Scattering (photon detection): UHV necessary
- X-ray-diffraction (in air)

Matter to be explored

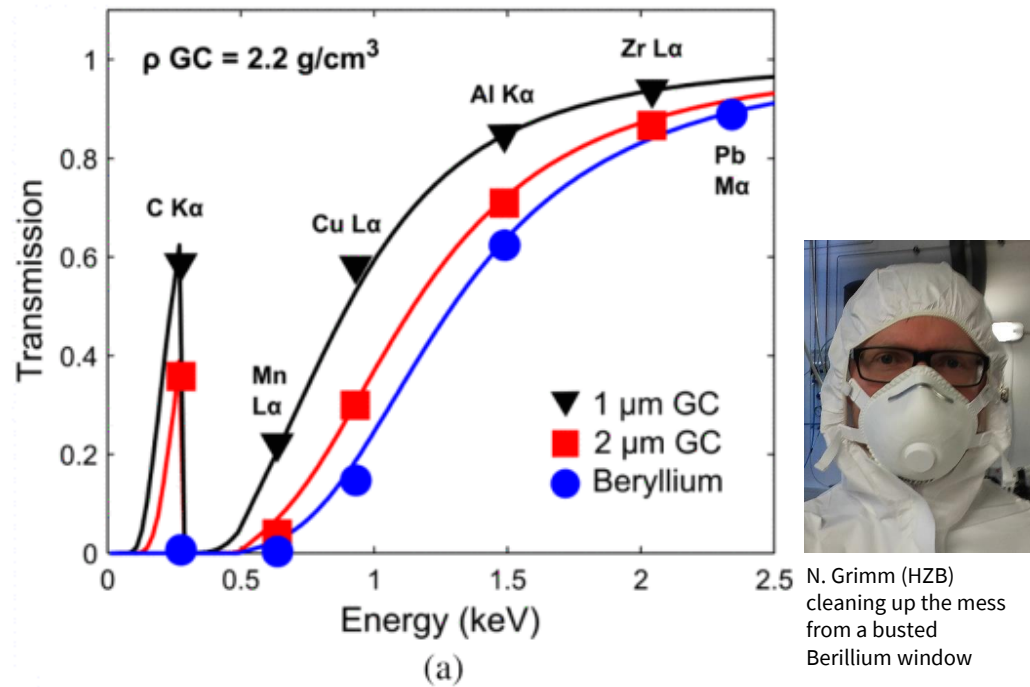
- Solids, Crystals
- Atoms, molecules, clusters
- Nanoparticles
- Liquids
- Surfaces



General challenges: windows

	Beryllium	Polymers (Kapton)	SiN	Graphenic Carbon	Quartz	Diamond	Aluminum
scattering properties							
Transparency (E)	+	++	++	++	+	++	+ >10 keV
coherent (Bragg) scattering	-	++	-	-	++	-	-
incoherent scattering							
physical properties							
mechanical strength	+	-	+/-	+/-	+	++	++
chemical resistivity	?	?			+		
thermal properties	conducting	insulating			insulating		conducting
electromagnetic properties							
construction suitability							
machinability	--	+	-	-	-	-	++
welding/saltering/possibilities	+	-	-	-	-	-	+
health aspects							
radioactivity							
toxicity	--						
other							
price	-	++	-	-	++	--	++
Typical Applications	"high" pressure windows					highest pressure windows	high pressure windows, low temperatures

General challenges: transmission of window materials



N. Grimm (HZB) cleaning up the mess from a busted Beryllium window

S. Huebner, et al. (2015). High Performance X-Ray Transmission Windows Based on Graphenic Carbon. Nuclear Science, IEEE Transactions on. 62. 10.1109/TNS.2015.2396116.

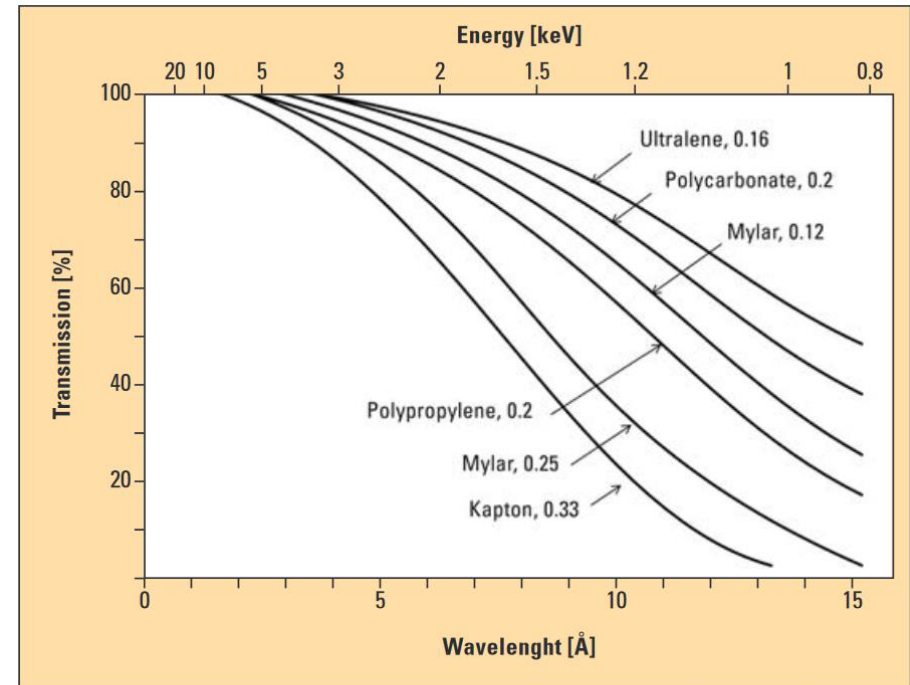
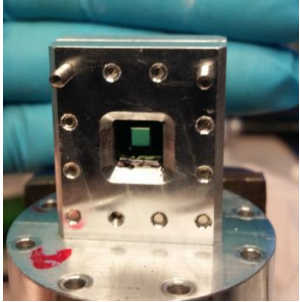


Figure 2: Transmission capacity of various polymer films as a function of the wavelength of the incident X-ray radiation. Lighter elements exhibit a higher wavelength and a lower energy. They are found at the right-hand side of the graphic.

Shimadzu News 1/2012

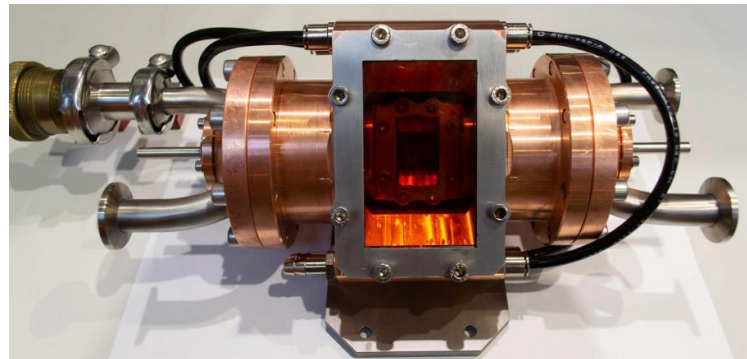


SiN – membrane (200nm) in gas adsorption cell for ASAXS and XAS, HZB



Kapton: miniature cryostat, HZB

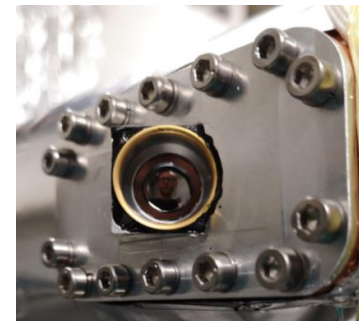
General challenges: windows



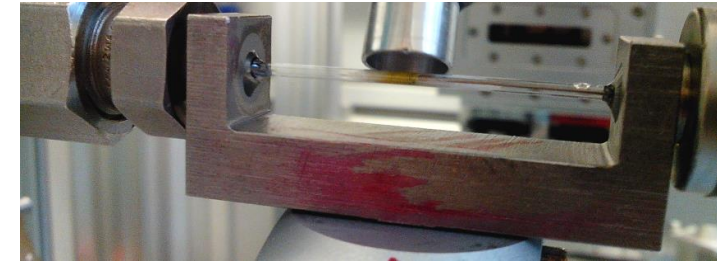
Kapton: vapor chamber, HZB



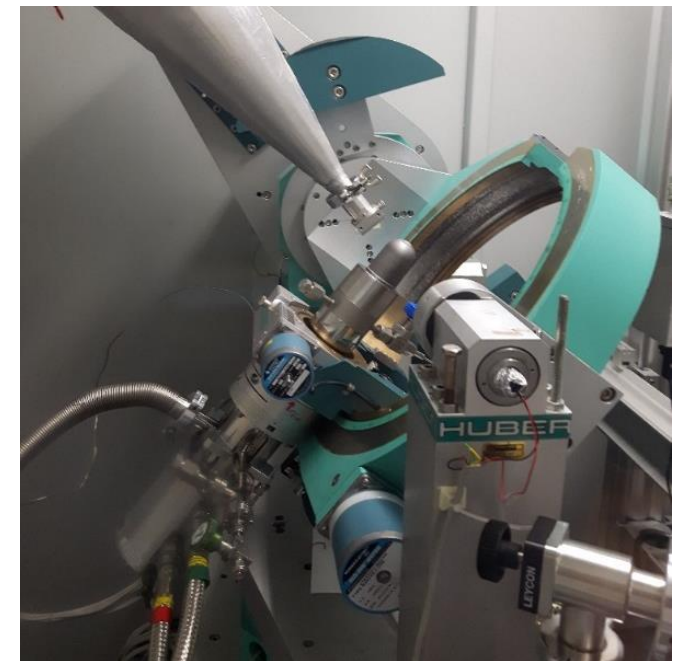
Ultralene membrane (4 μ) for Omicron compatible liquid sample cells, KMC-1, HZB



Graphenic carbon window for XES at PINK-beamline, HZB



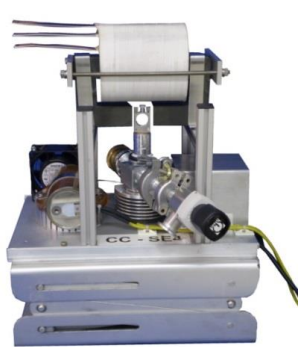
Quartz tubes: cryo- and hot air jets, KMC-2, HZB



Berillium: gas adsorption & cryostat, KMC-2, HZB

Sample Environment for T, B, p: temperature

- Low temperatures (closed cycle, Helium, Nitrogen, flow-cryostats, bath-cryostats, Peltier,...)
- Very low temperatures (^3He , $^3\text{He}/^4\text{He}$ dilution)



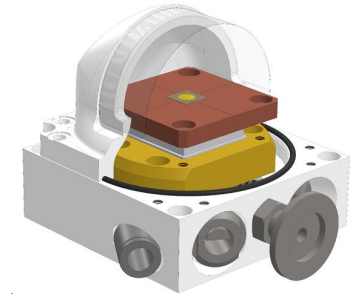
Miniature Stirling cooler (35-320 K)



Gifford McMahon cooler (4 - 600 K)



Automatic He-transfer for 7T horizontal cryomagnet:
High Field Diffractometer UE46_PGM-1, HZB



Mini Peltier sample stage for Bruker
D8 series (200K - 320K)



N-HeliX, OxfordCryosystems (T < 60 K)

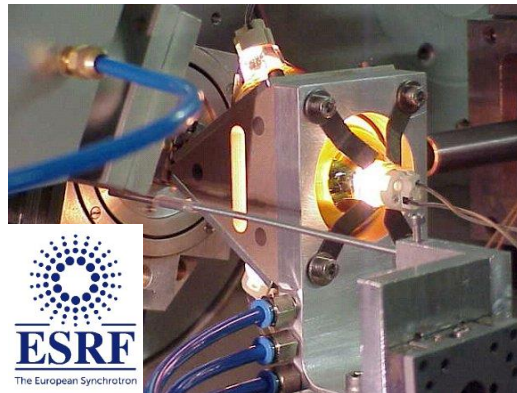


Sample Environment for T, B, p: temperature

- High temperatures (resistive, radiation, levitation, hot air blower, Peltier,...)

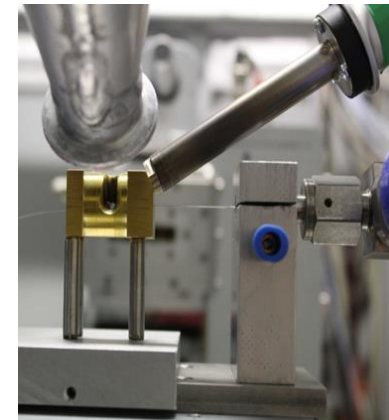


KMC-2 at BESSY-II: custom-built Beryllium dome furnace with double sided borolectric® heater reaches temperatures up to 1000 °C in vacuum (< 10⁻⁹ mbar)



Halogen-bulb **Mirror Furnace**. Sample contained in Pt capillary heated to ~1500°C (Andrew N. Fitch, ID22, ESRF)

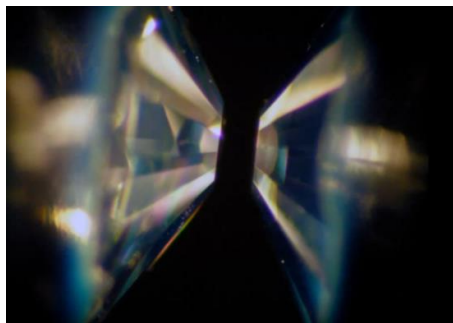
See as well (alternative design): Rev. Sci. Instrum. **91**, 065109 (2020); <https://doi.org/10.1063/1.5141139>



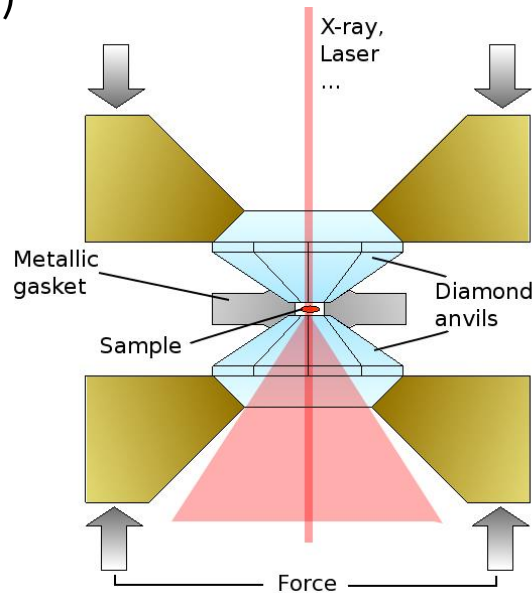
Hot-air-jet at KMC-2 beamline of BESSY-II
200-800 K

Sample Environment for T, B, p: pressure

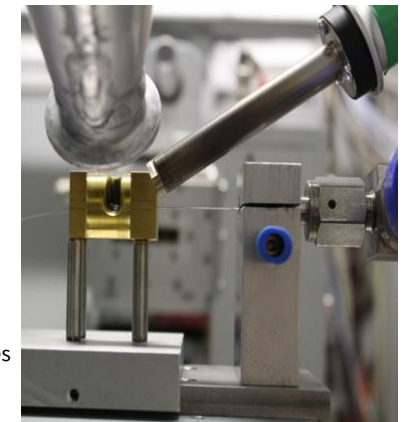
- Clamped cells (diamond anvil cells, some 100 GPa)
- Gas pressure (some 100 bar)
- Near ambient pressure (some 10 mbar)



Diamond anvils (D. Haase, MAX IV)



Sketch of a diamond anvil cell
Illustration by Sebastian Merkel, Lille



Gas adsorption in glass capillaries
up to 100 bar, 200 - 800 K
at KMC-2 beamline (BESSY-II)

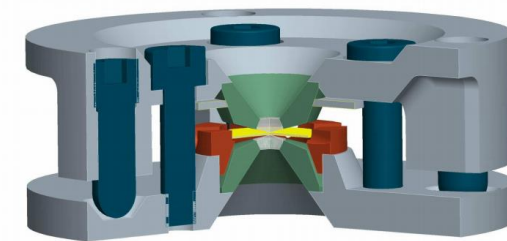


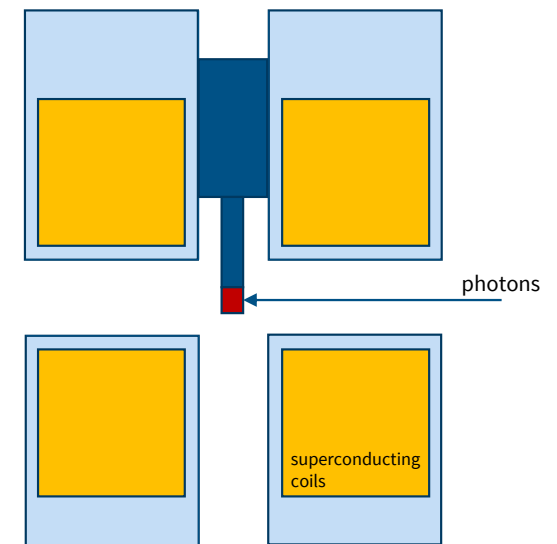
Figure 2: Cross section of Diacell® Bragg XVue.

DAC with side access for XRD (Illustration from Almax
easylab technical notes for Plate DAC)

Sample Environment for T, B, p: magnetic field

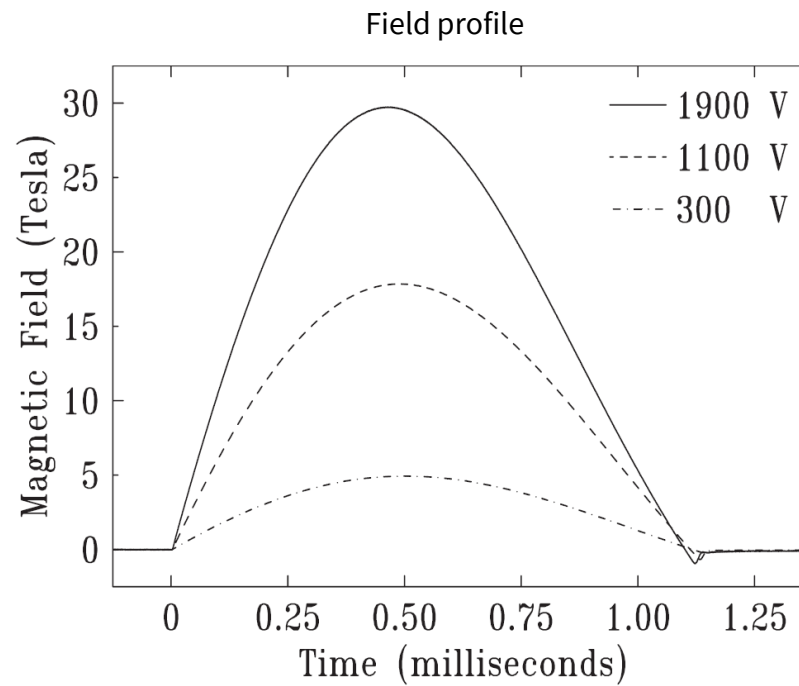
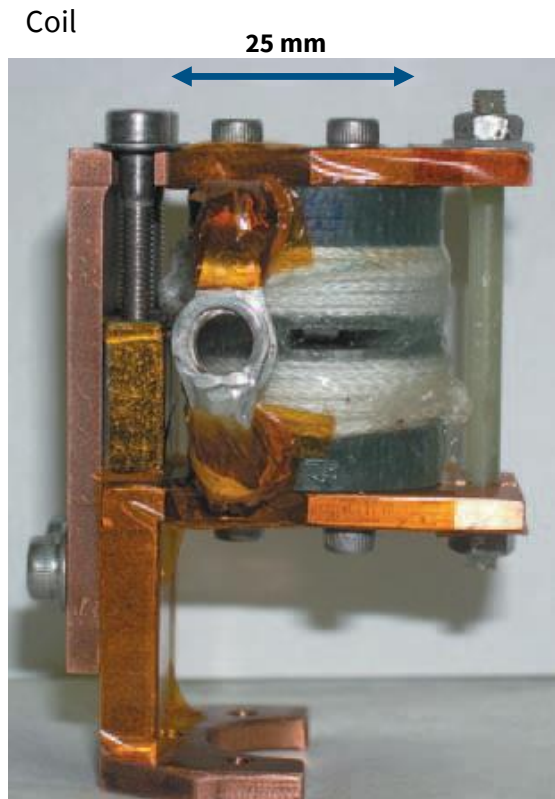
Vekmag at BESSY-II:

- 9T – 2T – 1T vector magnet
- different temperature inserts
- UHV sample space
- liquid Helium bath
- recondensing

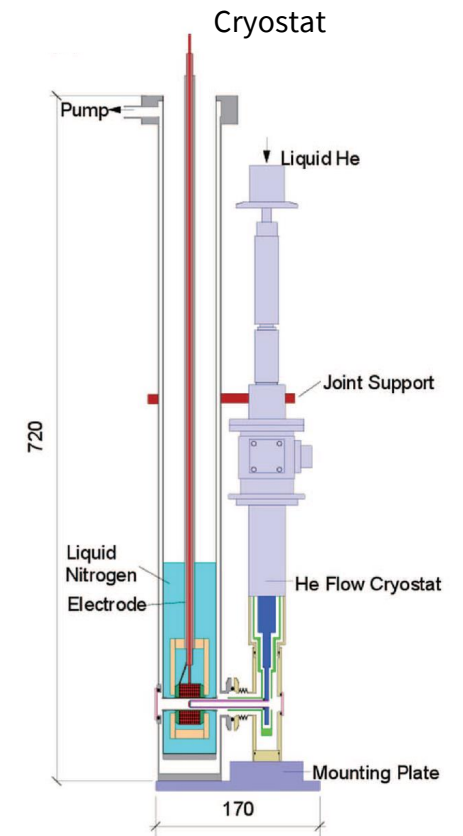


Split pair magnet

Sample Environment for T, B, p: pulsed magnetic fields

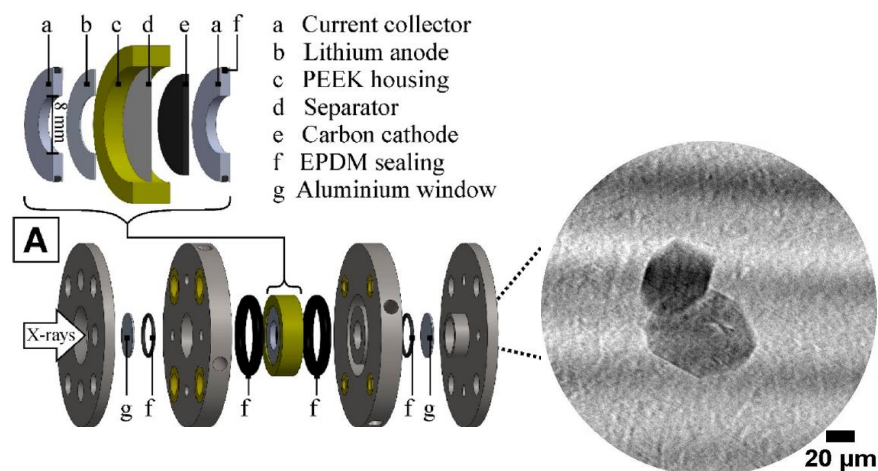


Z. Islam, J. Ruff, Hiroyuki Nojiri, et al., *Rev. Sci. Instrum.* **80**, 113902 (2009);
<https://doi.org/10.1063/1.3251273>
G. T. Noe, H. Nojiri, et al., *Rev. Sci. Instrum.* **84**, 123906 (2013);
<https://doi.org/10.1063/1.4850675>



In-situ, in-operando & other complex things

Battery research

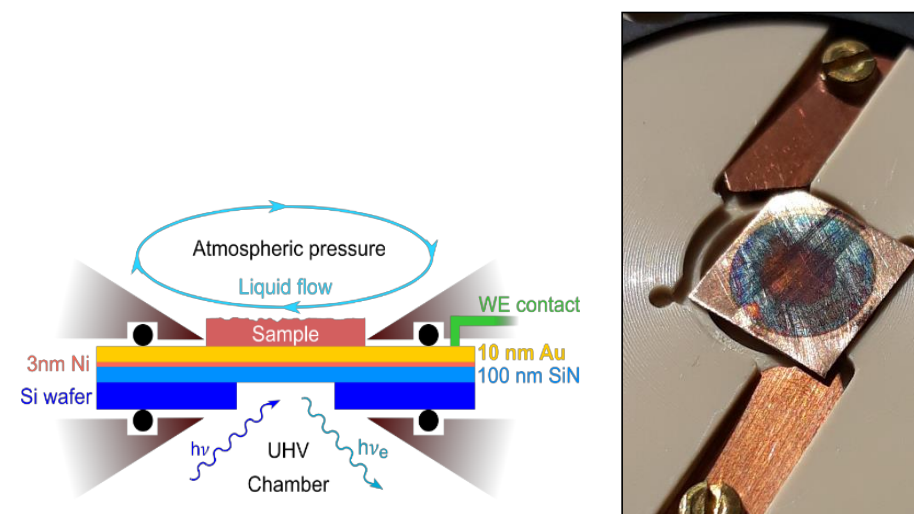


In-operando cells for battery research / X-ray Imaging
 S8 formation in lithium/sulfur batteries during cyclic voltammetry

S. Risse et al., *J. Phys. Chem. Lett.* **11** (2020) 5674–5679

Video: <https://doi.org/10.1021/acs.jpcllett.0c01284>

Electro chemical reactions



In operando electrochemical flow-through cell for XES / XAS (OÆSiS in EMIL@BESSYII)

- Electrochemistry with 3-electrode design
- Vacuum and liquid separated by 100 nm SiN membrane

D. Wallacher, R. Garcia Diez, M. Bär et al.

In-situ, in-operando & other complex things: liquids

Liquid jets et al.



Flat jet, HZB

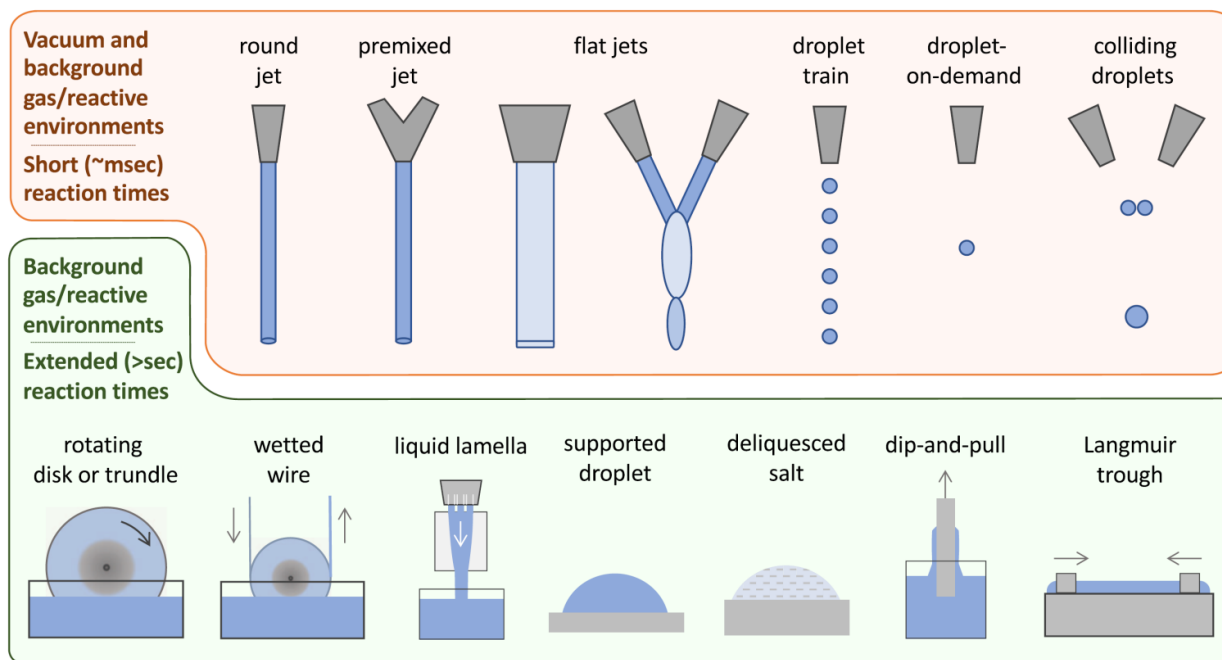
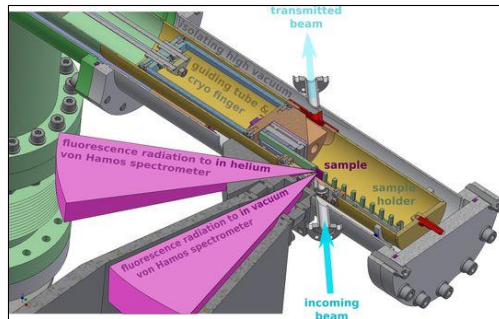


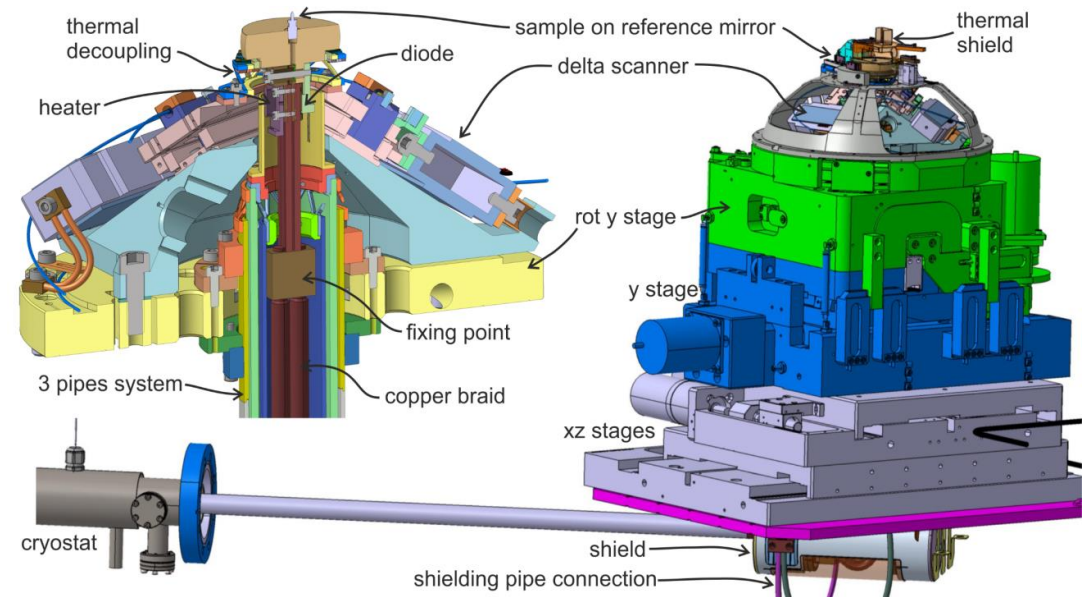
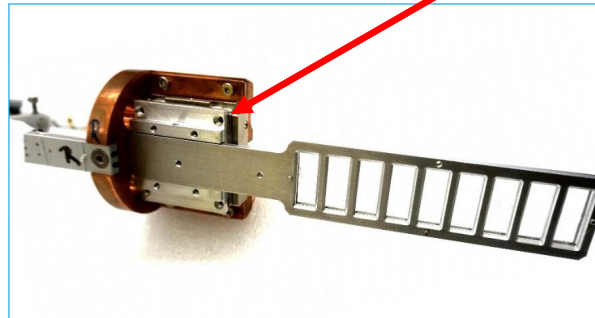
FIG. 4. Preparation of liquid–vapor interfaces inside vacuum chambers. The upper row shows methods based on fast moving jets or droplets that have a very brief interaction time with the surrounding environment before being measured. These methods can be used in a vacuum environment since the liquid reservoir is outside of the vacuum chamber. The bottom row shows preparation methods where the bulk reservoir of the liquid is inside the vacuum chamber and the measurements thus are done in the presence of the equilibrium vapor pressure. These investigations permit longer interaction times between gases and the liquid.

R. Dupuy, et al., J. Chem. Phys. **154**, 060901 (2021); doi: 10.1063/5.0036178

Sample positioning



attocube piezo actuator



PINK: Cryostat for X-Ray Emission Spectroscopy @ EMIL (HZB)

Temperature 20K .. 300 K

S. DeBeer, S. Peredkov, R. Ringel, K. Martin, L. Drescher, D. Wallacher, R. Grüneberger, B. Klemke, H. Thiel

Nanopositioning: OMNY at PSI

Tomography nano cryo endstation

Temperature 90K (LN2), 10 K (LHe)

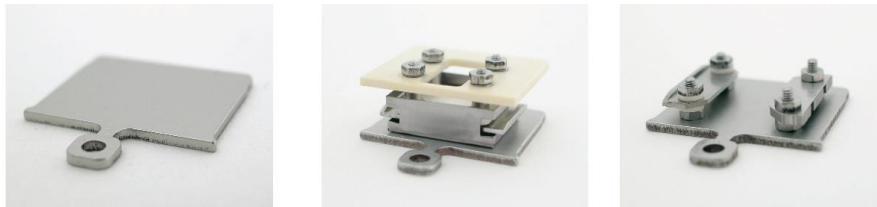
M. Holler, Jörg Raabe, et al., PSI

Review of Scientific Instruments **89**, 043706 (2018); <https://doi.org/10.1063/1.5020247>



Sample mounting: standardized flag style sample plates

Omicron

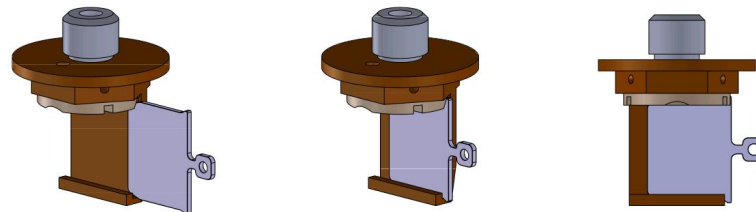


www.scientaomicron.com

Ferrovac



www.ferrovac.com



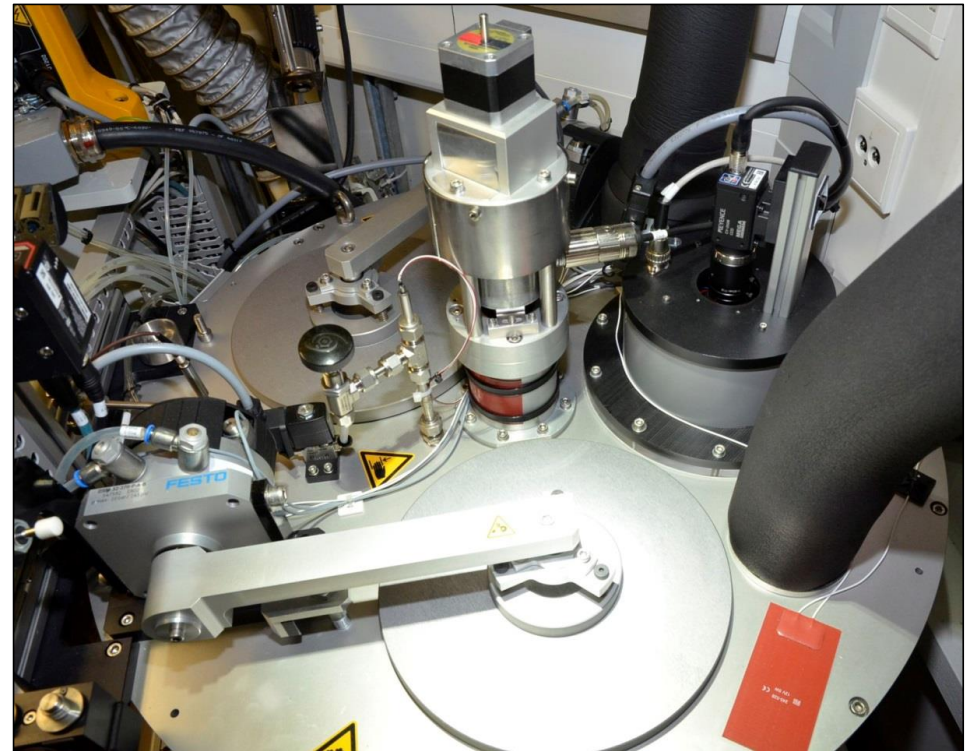
www.esrf.eu



CoESCA station, T. Leitner, HZB:
put sample into preparation chamber

Sample mounting: standardized sample holders for MX Macromolecular Crystallography

Example:
**Macromolecular
crystallography beamline
BL14.2 (HZB)**

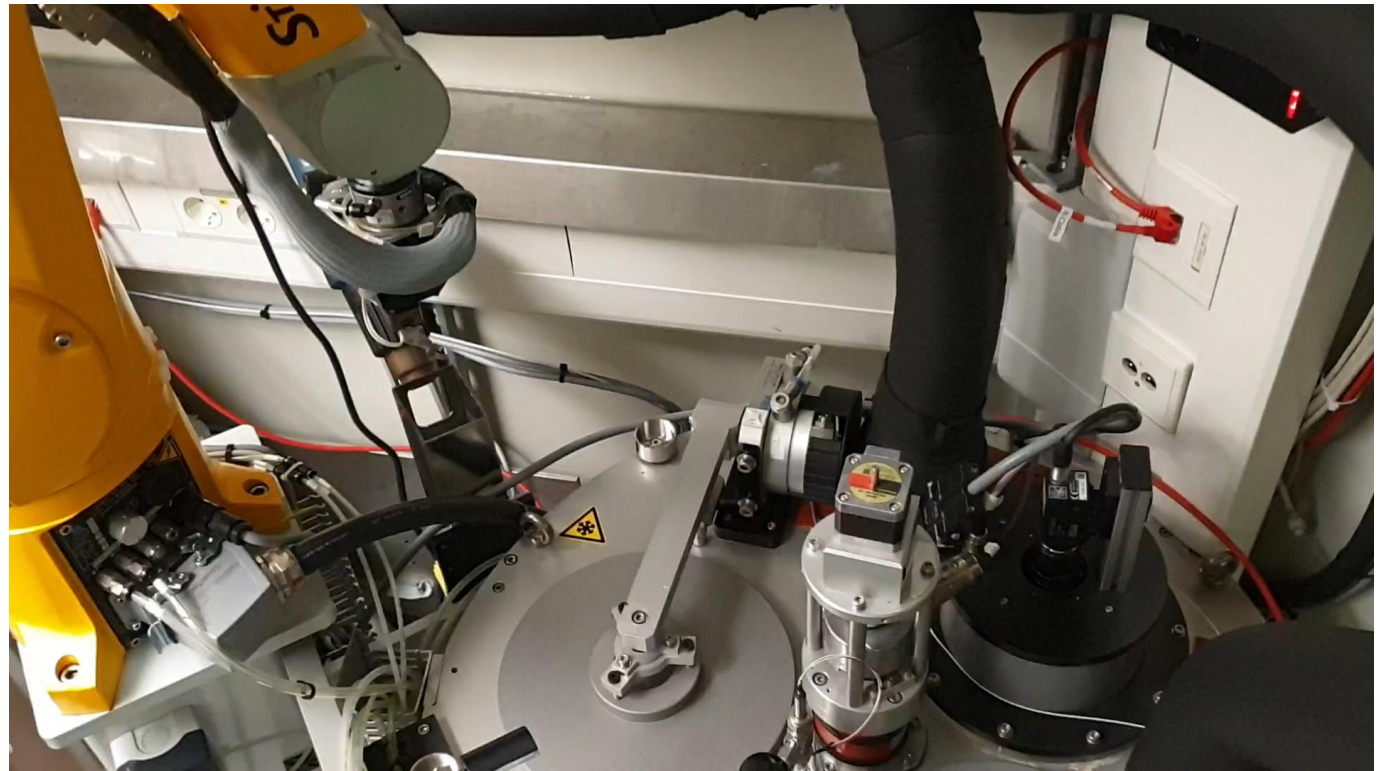


G-rob sample changer

U. Mueller et al. (2012). J. Synchrotron Rad. 19, 442-449
U. Mueller et al. (2016). Eur. Phys. J. Plus 130, 141-150

Sample mounting: standardized sample holders for MX Macromolecular Crystallography

Example:
**Macromolecular
crystallography beamline
BL14.2 (HZB)**



G-rob sample changer

Video by C. Feiler (HZB)

Conclusion (first part)

- Sample environment is crucial
- It is getting more complex
- Some things you just buy, others have to be customized or developed from scratch
- Standardisation and collaboration helps